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IS 1448 [P : 11] : 2004
ISO 3013 : 1997

भारतीय मानक

पैट्रोलियम और उसके उत्पादों की परीक्षण पद्धतियाँ

[पी : 11]

पैट्रोलियम उत्पाद – विमान ईंधन का जमाव बिन्दु ज्ञात करना

(चौथा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 11]

PETROLEUM PRODUCTS — DETERMINATION OF FREEZING POINT OF AVIATION FUELS

(Fourth Revision)

ICS 75.160.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2004 **Price Group 4**

Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

NATIONAL FOREWORD

This Indian Standard [P : 11] which is identical with ISO 3013 : 1997 'Petroleum products — Determination of the freezing point of aviation fuels' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

Third revision of this standard was published in 1990 which was based on ASTM D 2386 : 1967 and IP 16 : 1983. The Committee decided to revise this Indian Standard to completely align with ISO 3013 : 1997 and publish under the dual numbering system. Accordingly the title of the standard has been modified.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

CROSS REFERENCES

In this adopted standard reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their place, are listed below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO Standard, which has subsequently been revised, position in respect of that latest ISO Standard has been given:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3170 : 1988 Petroleum liquids — Manual sampling	IS 1447 (Part 1) : 2000 Petroleum and its products — Methods of sampling: Part 1 Manual sampling (*first revision*)	Technically equivalent with minor deviations
ISO 3171 : 1988 Petroleum liquids — Automatic pipeline sampling	Nil	—

In case of ISO 3171, the committee responsible for the preparation of this standard took cognizance of this Standard and decided that this is acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 11]

PETROLEUM PRODUCTS — DETERMINATION OF FREEZING POINT OF AVIATION FUELS

(Fourth Revision)

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a procedure for determining the temperature below which solid hydrocarbon crystals are present in aviation turbine fuels or aviation gasolines.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

3 Definitions

For the purposes of this International Standard the following definitions apply:

3.1 freezing point: The temperature at which solid hydrocarbon crystals, formed on cooling, disappear when the temperature of the test sample is allowed to rise.

3.2 crystallization point: The temperature at which crystals of hydrocarbons first appear when the test sample is cooled.

4 Principle

The sample tube, containing the test sample, a stirrer, collar and thermometer, is placed in a vacuum flask containing a coolant. During the cooling cycle the test sample is stirred vigorously and examined for the formation of crystals. When crystals are observed, the temperature is recorded as the crystallization point. The jacketed sample tube is then removed from the coolant and the test sample allowed to warm up slowly. Stirring continues until all crystals disappear, at which point the temperature is recorded as the freezing point.

5 Reagents and materials

5.1 Coolant

Use one of the following liquids specified in 5.1.1 to 5.1.4 in combination with solid carbon dioxide (5.1.5) or liquid nitrogen (5.1.6) under the specified conditions only.

5.1.1 Propan-2-ol [$(CH_3)_2CHOH$], commercial grade of dry propan-2-ol.

NOTE — Propan-2-ol is the preferred liquid for use as the coolant.

5.1.2 Ethanol (C_2H_5OH), commercial grade of dry ethanol.

5.1.3 Acetone (CH_3COCH_3), commercial grade, provided it does not leave a residue.

5.1.4 Methanol (CH_3OH), commercial grade of dry methanol.

5.1.5 Carbon dioxide (solid) or **"dry ice"**, commercial grade.

WARNING — Extremely cold, – 78 °C. Liberated gases can cause suffocation.

NOTE — Mechanical refrigeration is permitted provided that the coolant temperature is in the range – 70 °C to – 80 °C.

5.1.6 Nitrogen (liquid), commercial grade. Use only when determining the freezing point of aviation gasolines, or if the freezing point of the aviation turbine fuel is lower than – 65 °C.

WARNING — Extremely cold, – 196 °C. Liberated gases can cause suffocation.

5.2 Nitrogen gas or **dry air**, a supply of nitrogen gas of low moisture content or dry air shall be used if moisture-proof collar A is utilized.

5.3 Fibreglass, commercial grade, for use in moisture-proof collar B.

5.4 Dehydrating agent

Use one of the following:

5.4.1 Calcium sulfate ($CaSO_4$), granulated anhydrous calcium sulfate, for use as a desiccant in moisture-proof collar B, or to assist in drying the nitrogen gas or air (5.2) used with collar A.

5.4.2 Silica gel, 1,7 mm, for use as a desiccant in moisture-proof collar B, or to assist in drying the nitrogen gas or air (5.2) used with collar A.

6 Apparatus

See figure 1.

6.1 Jacketed sample tube, a double walled, unsilvered vessel, similar to a Dewar flask; the space between the jacketed sample tube and the outer glass jacket being filled, at atmospheric pressure, with dry nitrogen or air. The mouth of the tube shall be closed with a cork stopper supporting the thermometer and collar through which the stirrer passes.

6.2 Collar, as shown is either figure 2 or 3, and moisture-proof to inhibit the ingress of moisture into the test sample.

6.3 Stirrer, a brass or stainless steel rod, 1,6 mm in diameter, bent into a smooth three loop spiral at the bottom.

NOTE — The stirrer can be mechanically actuated.

6.4 Vacuum flask, an unsilvered vacuum flask of the dimensions shown in figure 1. The capacity shall be sufficient to hold an adequate volume of coolant (5.1) and permit the necessary depth of immersion of the jacketed sample tube.

6.5 Thermometer, conforming to annex A.

7 Apparatus preparation

7.1 Glassware

All glassware shall be clean and dry before use.

7.2 Collar

Assemble the collar (6.2), thermometer (6.5) and stirrer (6.3) into the cork.

7.2.1 Collar type A, flush with nitrogen or dry air (5.2) before fitting to the jacketed sample tube, and throughout the entire determination.

NOTE — The air can be effectively dried by passing through absorbent tubes filled with dehydrating agents (5.4.1 and 5.4.2).

7.2.2 Collar type B, fill with fibreglass (5.3) and a suitable dehydrating agent (5.4.1 or 5.4.2) as shown in figure 3. The fibreglass shall be replaced every fourth test. The dehydrating agent should be renewed at intervals of not more than 3 months or when a colour change shows it to be ineffective.

7.3 Vacuum flask

Insert sufficient coolant (5.1) to meet the immersion depth requirements shown in figure 1.

8 Samples and sampling

Obtain samples in accordance with ISO 3170, ISO 3171 or an equivalent national standard.

Dimensions in millimetres

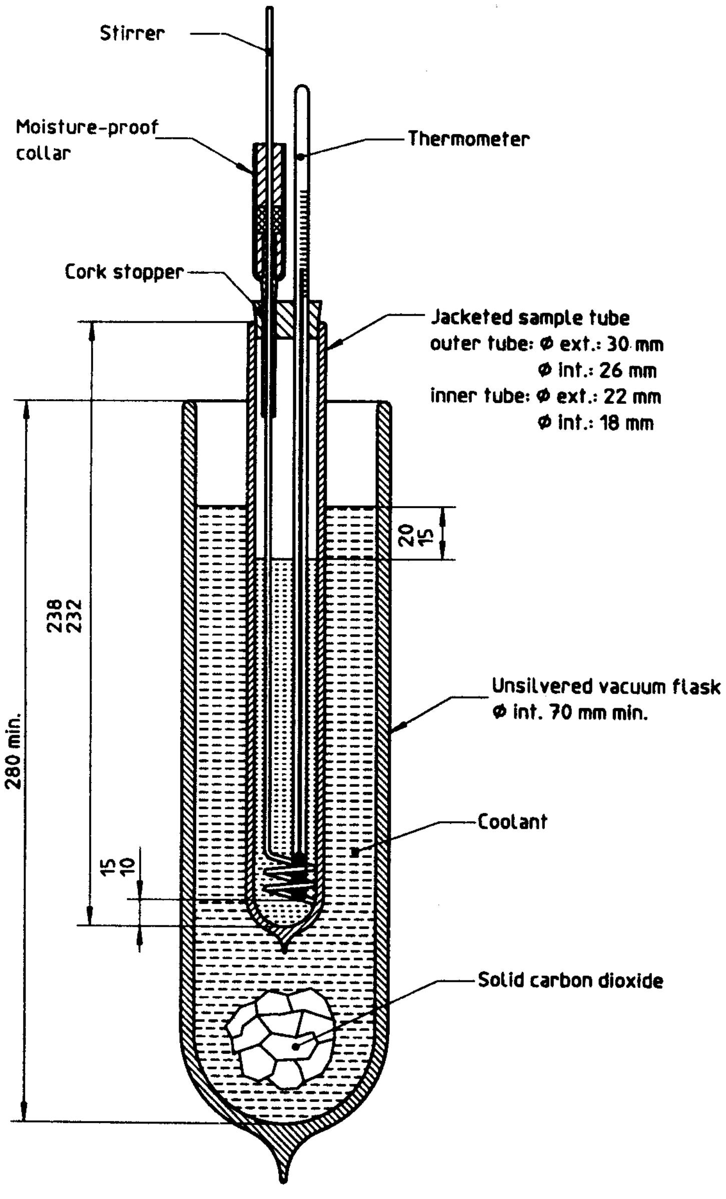

Figure 1 — Apparatus for determining the freezing point of aviation fuels

Dimensions in millimetres

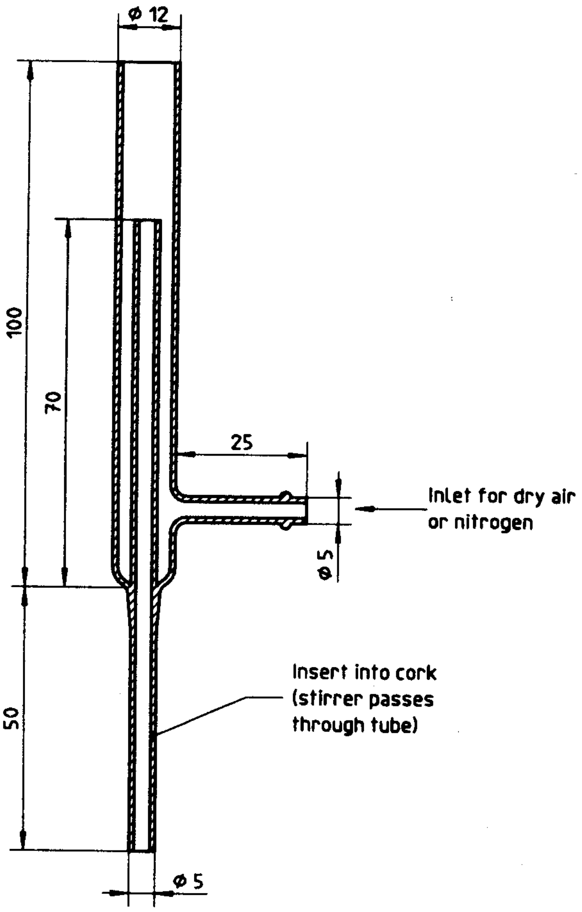
Ø 12
100
70
25
Inlet for dry air or nitrogen
Ø 5
Insert into cork (stirrer passes through tube)
50
Ø 5

Figure 2 — Moisture-proof collar type A

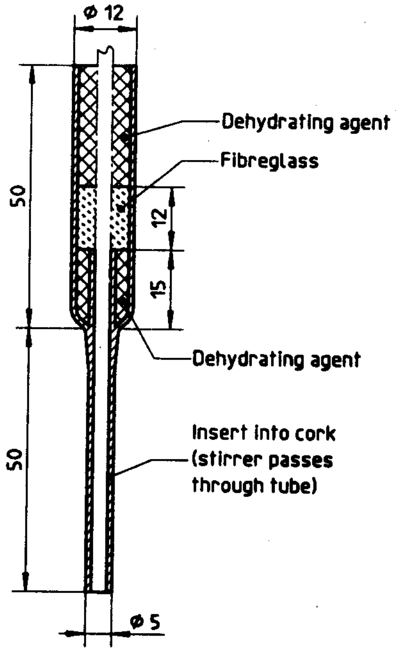

Figure 3 — Moisture-proof collar type B

9 Procedure

9.1 Transfer 25 ml ± 1 ml of test sample to the clean and dry jacketed sample tube (6.1). Close the tube tightly with the cork holding the stirrer, thermometer and collar; adjust the thermometer position so that its bulb is 10 mm to 15 mm above the centre of the bottom of the tube.

9.2 Clamp the jacketed sample tube so that it extends into the vacuum flask (6.4) containing the coolant. The surface of the test sample shall be approximately 15 mm to 20 mm below the level of the coolant. Add solid carbon dioxide (5.1.5) as necessary throughout the test to maintain the coolant level in the vacuum flask.

WARNING — Do not add solid carbon dioxide to liquid nitrogen.

9.3 Except when making observations, stir the test sample continuously, moving the stirrer up and down at a rate of between one cycle per second and 1,5 cycles per second. Stir the test sample from top to bottom, taking care that the stirrer loops do not make contact with the bottom of the jacketed sample tube or travel above the test sample's surface.

9.4 During the cooling cycle examine the test sample for the presence of crystals. Disregard any cloud that appears at approximately - 10 °C and does not increase in intensity as the temperature is lowered; this is due to water. However should such a cloud appear, note the observation in the report, specifying the temperature when the cloud first appeared and a description of its persistence.

When the formation of crystals is noted, record the temperature as the observed crystallization point.

NOTES

1 A diffused, cool, light source is recommended to assist in viewing the formation and disappearance of the crystals.

2 If it becomes difficult to view the appearance of crystals due to gases released by the coolant, the jacketed sample tube may be removed from the coolant for a period not exceeding 10 s. If crystals are found to have already formed, note the temperature of the sample and allow the sample to warm until it is 5 °C warmer than the temperature when the crystals are seen to disappear. The sample tube should then be replaced in the coolant and allowed to cool. The crystallization point may then be observed by removing the sample tube from the coolant when the temperature of the sample is slightly warmer than the noted temperature.

9.5 When the crystallization point has been determined, remove the jacketed sample tube from the coolant, continue stirring at a rate of between one cycle per second and 1,5 cycles per second, and allow the test sample to warm up slowly. Observe the test sample and when the crystals completely disappear, record the temperature as the observed freezing point.

10 Expression of results

Correct the observed freezing and crystallization points by applying the relevant thermometer correction. When the observed freezing or crystallization point is between two calibration temperatures, obtain the correction at the observed temperature by linear correlation. Report the corrected freezing point to the nearest 0,5 °C.

11 Precision

The precision of this method, as obtained by statistical examination of inter-laboratory test results, is as follows:

11.1 Repeatability, *r*

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, in the normal and correct operation of the test method, would exceed the following value in only one case in twenty.

$r = 0{,}8$ °C

11.2 Reproducibility, R

The difference between two single and independent results, obtained by different operators in different laboratories on nominally identical test material, in the normal and correct operation of the test method, would exceed the following value in only one case in twenty.

$R = 2{,}3$ °C

NOTES

1 The precision has been determined according to ISO 4259 except that the rounding rules have not been applied in this instance.

2 The sample matrix on which the precision of this standard is based did not include aviation gasoline.

12 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification the product tested;

c) the results of the test (see clause 10);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

Annex A
(normative)

Thermometer specification

A.1 The thermometer described in 6.5 shall meet the requirements given in table A.1.

Table A.1 — Requirements for the thermometer for the determination of the freezing point of aviation fuels

Range	– 80 °C to + 20 °C
Immersion	Total
Graduations at each	0,5 °C
Longer lines at each	1 °C and 5 °C
Figured at each	5 °C
Scale error not to exceed	1 °C
Expansion chamber permitting heating to	45 °C
Overall length	300 mm ± 5 mm
Stem diameter	6,0 mm to 8,0 mm
Bulb length	8 mm to 16 mm
Bulb diameter	no greater than stem
Bulb shape	cylindrical
Length of graduated portion	170 mm to 210 mm
Distance from bottom of bulb to 0 °C line	220 mm maximum
Top finish	plain or ring
Liquid colour	permanent red
Filling above liquid	gas under pressure

NOTES

A.1 Thermometer type IP 14C/ASTM 114C meets the above requirements.

A.2 Toluene is suitable as the actuating liquid.

A.3 The thermometer shall have a valid calibration certificate.

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